LSASD Project ID: 20-0018

## Sample and Analysis Plan

# **Characterization of Ambient PFAS in the Chattooga River Watershed**

Project Location: Chattooga River Watershed (Georgia and Alabama)

**Project Date(s):** November  $4^{th} - 8^{th}$ , 2019

Final SAP Approval Date: October 25, 2019

**Project Leader: Greg White** 

Environmental Sampling Section Applied Science Branch Laboratory Services & Applied Science Division USEPA – Region 4 980 College Station Road Athens, Georgia 30605-2720

The activities depicted in this Sampling and Analysis Plan (SAP) are accredited under the US EPA Region 4 Laboratory Services & Applied Science Division ISO/IEC 17025 accreditation issued by the ANSI-ASQ National Accreditation Board. Refer to certificate and scope of accreditation AT-1644.





SESDFORM-064-R1

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#### **Analytical Support:**

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LSASD Project Leader:

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This Sample and Analysis Plan (SAP) is designed to be used in conjunction with the Applied Science Branch Quality Assurance Project Plan (USEPA, 2019a).

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SECTION A: Project Planning Elements					
A1. Distribution List					
Recipient	Organization	Address/Email			
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	A2. Project Personnel				
Team Members <sup>1,2</sup>	Organization	Responsibilities			
Greg White	EPA/R4/LSASD	Project Leader			
Nate Barlet	EPA/R4/LSASD	Safety Officer/Sampler			
Jerry Ackerman	EPA/R4/LSASD	Discharge Task Lead/Sampler			
Mel Parsons	EPA/R4/LSASD	Sampler			
John Ruiz	EPA/R4/LSASD	Sampler			
Bill Simpson	EPA/R4/LSASD	Sampler			

<sup>&</sup>lt;sup>1</sup> Project team members subject to change due to scheduling conflicts.

<sup>&</sup>lt;sup>2</sup> Project Leader and all Task Leaders assisting with this project have been deemed competent by LSASD management, under ISO 17025 accreditation, to conduct the tasks required to fulfill the prescribed goals.

#### A3. Site Description and Background Information

The headwaters of the Chattooga River begin in Walker County north of LaFayette Georgia. The Chattooga River flows south across the Alabama-Georgia state line and feeds into Weiss Lake near Gaylesville Alabama. Per- and polyfluoroalkyl substances (PFAS), specifically perfluorooctanesulfonate (PFOS), were detected in the surface water at several sites along the Chattooga River during a 2018 study performed by U.S. EPA Region 4's Laboratory Services & Applied Science Division (LSASD) (USEPA, 2018). PFAS have also historically been detected downstream at public drinking water intakes in Centre Alabama and Gadsden Alabama (USEPA, 2019b).

PFAS are man-made chemicals that do not occur in nature and have been found to be persistent and accumulate in both the environment and the human body via exposure pathways such as consumption of contaminated food and drinking water. PFAS have been extensively used in industry, manufacturing of commercial products, and most notoriously as a component in aqueous film forming foams (AFFF) used for firefighting. There is evidence that suggests exposure to PFAS can lead to adverse health effects and are an emerging concern to public health. PFAS is a generic nomenclature encompassing a broader array of chemicals, with the most studied being perfluoroctanoic acid (PFOA) and perfluoroctanesulfonate (PFOS). The U.S. EPA has issued a Recommended Health Advisory for drinking water of 70 ng/L (ppt) for combined concentrations of PFOA and PFOS compounds. Extensive information regarding PFASs can be found at <a href="http://www.epa.gov/pfas">http://www.epa.gov/pfas</a>.

#### A4. Problem Definition

The 2018 Phase 1 Study by LSASD observed positive detections of PFOS in surface water in the upper Chattooga River Watershed. PFOA was not detected in the 2018 study conducted by LSASD. The 2018 sampling of the Chattooga River by LSASD was conducted during an extreme high flow event thus dilution effects may have been a factor (USEPA, 2018). Major findings of the 2019 Phase 2 Study by LSASD with respect to the Chattooga River include: 1) the highest total PFAS concentrations of all streams sampled; 2) the highest diversity of distinct PFAS compounds detected; 3) a contribution of about 25% of total PFAS loading to Weiss Lake at a flowrate 7 times lower than the Coosa River during the study period (USEPA, 2019b). As guided by findings of the 2018 Chattooga River Study (Phase 1), the 2019 Weiss Lake Study (Phase 2), and directives of the R4 Water Division, this study will target near-base flow conditions as background concentrations of PFAS for the Chattooga River Watershed are largely unknown.

This study will observe background concentrations of PFAS in surface water and co-located sediment samples to characterize the relative distribution of PFAS in the Chattooga River Watershed along key segments determined by the Water Division. Ambient surface water concentrations of PFASs will be coupled with flow measurements to calculate instantaneous mass loading rates at near-base flow conditions. Sampling locations bracket off the following key segments of the Chattooga River based on the following municipalities in Georgia: Lyerly, Summerville, Trion, and LaFayette.

#### A5. Project Description, Goals, and Study Boundaries

#### Study Goal:

Characterize the distribution and instantaneous mass loading of PFAS in the Chattooga River Watershed at near-base flow conditions along key segments determined by the R4 Water Division.

#### Study Objectives:

- 1. Collect surface water samples coupled with discharge measurements to compute instantaneous mass loading rates of PFAS along key segments of the Chattooga River Watershed.
- Collect sediment samples collocated with surface water sample locations to determine the relative distribution and the potential for migration of PFAS contaminated sediments to the receiving waters of Weiss Lake.

#### Study Area:

The study area for this project includes the main stem of the Chattooga River and several inflowing tributaries. Proposed sampling locations range from the lower Chattooga River near Gaylesville Alabama where the river terminates into Weiss Lake, to the headwaters of the Chattooga River north of LaFayette Georgia (Appendix A: Site Maps). A total of 13 sites will be assessed which includes 8 stations on the main stem of the Chattooga River, 1 station on Town Creek which forms the headwaters of the Chattooga River, 3 tributary stations in watersheds with active biosolids application sites, and 1 station on Mill Creek, a significant tributary in Alabama. See Table 1 for a description of all sampling sites.

#### Study Design/Approach:

Standard Operating Procedures for all sampling and field measurement activities outlined in this study plan are referenced in Section B5: Sampling and Measurement Procedures.

#### In-Situ Water Quality

Surface water quality measurements of temperature, dissolved oxygen, specific conductance, turbidity, and pH will be collected *in-situ* via multi-parameter data sondes at each site. See Table 2 for a detailed list of *in-situ* water quality parameters and measurement uncertainties. All multi-parameter data sondes will be maintained and calibrated in accordance with LSASD Standard Operating Procedure for Equipment Inventory and Management (SESDPROC-1009-R0) and those selected in Section B.5. All equipment calibrations will be verified in accordance with LSASD Calibration and End-Check Acceptance Criteria (SESDFORM-060-R0).

PFAS Loading Rates (Surface Water Sampling and Discharge Measurements)

Surface water samples will be collected at each site and transported to the EPA R4 Laboratory at LSASD in Athens Georgia to be analyzed for the 23 PFAS analytes listed in Table 4. PFAS sample collection, preservation, and holding times are listed in Table 3. Surface water sample collection methods will vary based on site conditions (e.g. direct fill method where possible or grab method via stainless-steel bucket or

scoop). A corresponding discharge will be either directly measured via handheld or remotely-operated flowmeters or retrieved from USGS gaging stations for each sampling location to compute an instantaneous mass loading rate of detected PFAS compounds. This study will target near-base flow conditions. Approximate base flow conditions will be defined as a discharge below the monthly mean for November as recorded by historical streamflow data collected at the USGS gage located on the lower Chattooga River (USGS 02398300) over a 30-year period. The threshold value is 450 ft<sup>3</sup>/s. A discharge above this threshold will be considered non-base flow conditions and the sampling event will be postponed until water levels recede to appropriate levels. Best professional judgement will be used in consultation with the R4 Water Division to determine how to proceed if these environmental conditions are not met.

#### Sediment Sampling

Sediment samples will be collected at each site and transported to the EPA R4 Laboratory at LSASD in Athens Georgia to be analyzed for the 23 PFAS analytes listed in Table 4. PFAS sample collection, preservation, and holding times are listed in Table 3. Sediment sample collection methods will vary based on site conditions (e.g. grab method via stainless-steel scoop where possible or an alternate grab method via stainless-steel petite Ponar). Each sediment sample will consist of a composite of 3 sediment aliquots collected across a transect perpendicular to the stream flow to account for streambed heterogeneity.

#### Quality Control Samples

Multiple control samples will be collected in accordance with LSASD Standard Operating Procedures and accepted trace-level contaminant sampling practices. Control samples will include trip blanks, field blanks, field equipment rinse blanks, field duplicate samples, and matrix spike/matrix spike duplicate (MS/MSD) field samples. Surface water and sediment samples collected for PFAS analysis will be sampled via a trace level sampling technique to avoid cross-contamination of PFAS samples due to sample collection and handling. This process will require two field personnel for PFAS sample collection. A designated sampler will handle the sample media and sample container only. A second designee will operate sampling equipment and assist with sample container packaging and labeling. An outline of all quality control samples is listed in Section B3: Quality Control.

#### Project Timeline:

All field activities for this study are planned for the week of November 4<sup>th</sup>, 2019. Laboratory turn-around time is 35 days from the time samples are received. The draft final report for this study is expected to be provided to the Water Division on January 22<sup>th</sup>, 2020.

### A6. Applicable Regulatory Information

The U.S. EPA has established a life-time exposure recommended health advisory level for drinking water of 70 parts per trillion for PFOA and PFOS individually or combined. There are currently no Maximum Contaminant Levels (MCLs) or enforceable standards for PFOA, PFOS, or other PFAS related compounds in any media (e.g. drinking water, surface water, soils and sediments) set by the U.S. EPA or the states of Georgia and Alabama.

#### A7. Decision(s) to be made based on data

This study will provide relative mass loadings of PFAS compounds in surface water along key segments of the Chattooga River Watershed and insight into the relative distribution and the potential for migration of PFAS compounds in sediments to the receiving waters of Weiss Lake; as well as provide a comparison of PFAS concentrations and compositions in sediment and surface water along the Chattooga River Watershed. All further decisions, recommendations, and/or actions will be made at the discretion of the U.S. EPA's R4 Water Division.

SE	CCTION B: Data Gen	eration,	Acquisition, and Reporting		
Will samples or phy	B1. Sampling  ample Media  Total Number of Sam  13 samples + duplicate + MS/MSD  Sediment  13 samples + duplicate + MS/MSD  B2. Samplin  outlined in the Applied Science Branch Quality dled and custody maintained in accordance wire Quality Assurance Manual, LSASD Operating, and LSASD Operating Procedure for Packing	⊠ Yes –	If yes, complete all subsections in Section B.		
· · · · · · · · · · · · · · · · · · ·		□ No -	If no, no action needed for B1, B2, B3 or B4,	proc	reed to B5.
	B1. Sampling	Design/I	nformation Inputs		
Sample Media	Total Number of San	ıples	Analyses		
Surface Water	13 samples + duplicate + + MS/MSD	+ 4 QC	PFAS (See Tables 3 & 4)		
Sediment	13 samples + duplicate + N	MS/MSD	PFAS (See Tables 3 & 4)		
handled and custody and Quality Assurance 005, and LSASD Ope SESDPROC-209.	plied Science Branch Quality maintained in accordance wi ce Manual, LSASD Operating erating Procedure for Packing	v Assurance th the LSA g Procedure	SD Laboratory Services Branch Laborators for Sample and Evidence Management,	ry O SES	perations DPROC-
Will a Chai	n-of-Custody be produced?				
Custody of a sample or  It is in the actu  It is in the view  It was in the p	physical evidence is defined as. ual possession of an investigator w of an investigator, after being	: r in their phy.	le to ensure that custody is maintained?  sical possession  en they secured it to prevent tampering		Yes

#### **B3. Quality Control**

Field quality control measures will be performed in accordance with the LSASD Operating Procedure for Field Sampling Quality Control, SESDPROC-011.

Field quality control (QC) samples include the following:

- Each batch of samples will contain a duplicate quality control sample for each analysis. The duplicate samples will be collected at CHR01.
- Each batch of surface water and sediment samples being analyzed for PFAS will also contain an additional sample volume for matrix spike/matrix spike duplicates (MS/MSD). MS/MSD volumes will be collected at TOC01 at the headwaters of the Chattooga River to characterize background PFAS concentrations in surface water and sediment.
- Temperature blanks will be placed in all sample coolers.

The following additional quality control (QC) samples will be collected and analyzed for PFAS contamination:

- A field blank will be collected by the sampling team at the onset and completion of field activities.
- Trip blank(s) will be stored and transported with collected samples through the duration of the study.
- If a field equipment decon is needed, a separate field equipment rinse blank will be collected for PFAS sediment sampling equipment (e.g. stainless-steel petite Ponar sediment grabs, spoons, scoops, and bowls), and PFAS surface water sampling equipment (e.g. buckets and/or scoops).
- All blank quality control (QC) samples will be prepared utilizing PFAS-free water supplied by the U.S. EPA LSASD laboratory in Athens, GA.

#### PFAS sampling protocol:

- A two-person trace-level sampling protocol will be used for all PFAS sample collection. One member of the sampling team will handle the sample media and sample container only. A second team member will be designated to handle sampling equipment and assist with sample packaging and labeling.
- All sampling equipment will be cleaned using Luminox® and warm tap-water, then rinsed in PFAS-free water before being air-dried and sealed in clean plastic sheets in preparation for field activities.
- Sampling equipment known to contain PFAS will be avoided during sampling activities.

Laboratory quality control measures are specified in the LSASD Laboratory Services Branch Laboratory Operations and Quality Assurance Manual (USEPA, 2019c).

⊠ No

□ Unknown

## **B4.** Analytical Methods and Support Samples will be analyzed by the EPA/LSASD laboratory in Athens, GA in accordance with the LSASD Laboratory Services Laboratory Operations and Quality Assurance Manual (USEPA, 2019c). Specific analytical methods are listed in Table 4. Samples submitted to a Contract Laboratory Program (CLP) laboratory will be analyzed in accordance to the current statement of work. Laboratory Turn-Around-Time Requested: Days $\boxtimes$ Non-Routine Reporting Levels ARE NOT Required, No Further Action. **Reporting Levels:** Non-Routine Reporting Levels ARE Required, List Below. Non-Routine n/aReporting Levels: □ Yes

Waste Samples Anticipated:

If answer is yes, specify laboratory to receive samples: n/a

#### **B5. Sampling and Measurement Procedures**

Sampling and measurement activities will be in accordance with the LSASD operating procedures. The following field procedures will be followed during this study, check all that apply. The most recent version of LSASD operating procedures can be found at <a href="https://www.epa.gov/quality/quality-system-and-technical-procedures-sesd-field-branches">https://www.epa.gov/quality/quality-system-and-technical-procedures-sesd-field-branches</a> (Last Update: 4/05/18)

Fiel	d Measurement Procedures*	SESDPROC-	Revision
×	Field pH Measurement	100	R4
×	Field Specific Conductance Measurement	101	R6
×	Field Temperature Measurement	102	R5
×	Field Turbidity Measurement	103	R4
	Groundwater Level and Well Depth Measurement	105	R3
×	Field Measurement of Dissolved Oxygen	106	R4
	Field X-Ray Fluorescence (XRF) Measurement	107	R4
	Wastewater Flow Measurement	109	R4
×	Global Positioning System	110	R4
×	In-Situ Water Quality Monitoring	111	R4
	Field Measurement of Total Residual Chlorine	112	R5
	Field Measurement of Oxidation-Reduction Potential (ORP)	113	R2
Fiel	d Sampling Procedures*	SESDPROC-	Revision
×	Sediment Sampling	200	R3
×	Surface Water Sampling	201	R4
	Soil Sampling	300	R3
	Groundwater Sampling	301	R4
	Waste Sampling	302	R3
	Ambient Air Sampling	303	R5
	Potable Water Supply Sampling	305	R3
	Wastewater Sampling	306	R4
	Soil Gas Sampling	307	R3
	logy Section Field Sampling Procedures*	SESDPROC-	Revision
×	Hydrological Studies	501	R4
	Water Column Oxygen Metabolism	504	R4
	Reaeration Measurement by Diffusion Dome	505	R4
	Sediment Oxygen Demand	507	R4
	Multi-Habitat Macroinvertebrate Sampling in Wadeable Freshwater Streams	508	R4
	Marine Macroinvertebrate Field Sampling	511	R4
	Fish Field Sampling	512	R4
	Pore Water Sampling	513	R3
	Dye Tracer Measurements	514	R2
	Bottom Water Sampling for Sulfide	515	R0

<sup>\*</sup>If procedures allow for different sampling and measurement methods, the utilized method(s) must be identified in the project description section. Additionally, verify procedure revision numbers before issuance of SAP.

Section C	: Reporting
C1. Re	eporting
Estimated Report Completion Date: 01/22/2020	
	⊠ Yes
Is a Provisional Data Release Anticipated?	□ No
and the analytical data have been released as final from the LS	a final field investigation report. Provisional data may be by if LSASD management approves the release of the information SASD Laboratory Services Branch, for LSASD generated data, generated data. Release of provisional data will be transmitted the Section Chief in accordance with the LSASD Operating
Additional Comments: Provisional data may be released report for the purpose of planning regional priorities rele	1 0 00

#### References

- SESDFORM-060-R0 (2018). SESD Calibration and End-Check Acceptance Criteria. U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.
- SESDPROC-1009-R0 (2017). Standard Operating Procedure for Equipment Inventory and Management. U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.
- USEPA (2018). Phase 1: Study of PFAS Compounds on the Chattooga River (Project ID 18-0142). U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.
- USEPA (2019a). Applied Science Branch Quality Assurance Project Plan. U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.
- USEPA (2019b). Phase 2: Prioritization of PFAS Contributions to Weiss Lake (Project ID 19-0253). U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.
- USEPA (2019c). Laboratory Services Branch Laboratory Operations and Quality Assurance Manual. U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.

**Table 1:** Sampling Site Locations and Descriptions

Station ID	Water Body	Coor	oximate dinates ldddd)	Site Description	
		Latitude	Longitude		
CHR01	Chattooga River	34.26362	-85.56017	Chattooga River at Hwy 35 in Gaylesville AL	
MIC01	Mill Creek	34.29581	-85.50949	Mill Creek at Hwy 68 near Gaylesville AL	
CHR02	Chattooga River	34.33585	-85.44564	Chattooga River at Rte 323 in Chattoogaville GA	
HIC01	Hinton Creek	34.33456	-85.43668	Hinton Creek at Rte 323 in Chattoogaville GA	
CHR03	Chattooga River	34.40220	-85.39595	Chattooga River at Lyerly Dam Rd in Lyerly GA	
CHR04	Chattooga River	34.44476	-85.36263	Chattooga River at Hwy 100 near Summerville GA	
RAC01	Raccoon Creek	Redacted	Redacted	Raccoon Creek upstream of Summerville public drinking water intake facility GA	
CHR05	Chattooga River	34.51955	-85.30120	Chattooga River at Penn Bridge Rd near Trion GA	
CHR06	Chattooga River	34.54532	-85.31792	Chattooga River upstream of low-head dam near Trion GA	
TEC01	Teloga Creek	34.54353	-85.38531	Teloga Creek at Hwy 327 in Broomtown Valley GA	
CHR07	Chattooga River	34.66671	-85.30005	Chattooga River at Foster Mill Dr near LaFayette GA	
CHR08	Chattooga River	34.70723	-85.28696	Chattooga River near Culberson Ave in LaFayette GA	
TOC01	Town Creek	34.71414	-85.26769	Town Creek at Round Pond Rd near LaFayette GA	

**Table 2:** *In-Situ* Water Quality Parameters

In-Situ Water Quality Parameter Measurement Uncertainty					
Parameter	Units	Measurement Technology	Measurement Uncertainty		
рН	SU	Glass electrode	± 0.2 SU		
Dissolved Oxygen	mg/L	Luminescent DO probe	± 0.2 mg/L		
Temperature	°C	LDO Thermistor	± 0.2 °C		
Specific Conductance	μS/cm	Nickel electrode cell	$\pm$ 0.5% of reading		
Turbidity	FNU	Optical Probe	± 5% of reading		

**Table 3:** Sample Collection, Preservation and Holding Times

Analyses	yses Media Container		Preservation	Holding Time
PFAS	Surface Water	2 x 15mL Polypropylene Vial	Ice (≤ 4°C)	42 days
	Sediment	50mL Polypropylene	Ice (≤4°C)	42 days

**Table 4: PFAS Target Analyte List** 

## Region IV Laboratory Per - and Polyfluoroalkyl Substances (PFAS) Target Analyte List Method Detection Limits (MDLs) & Minimum Reporting Limits (MRLs)

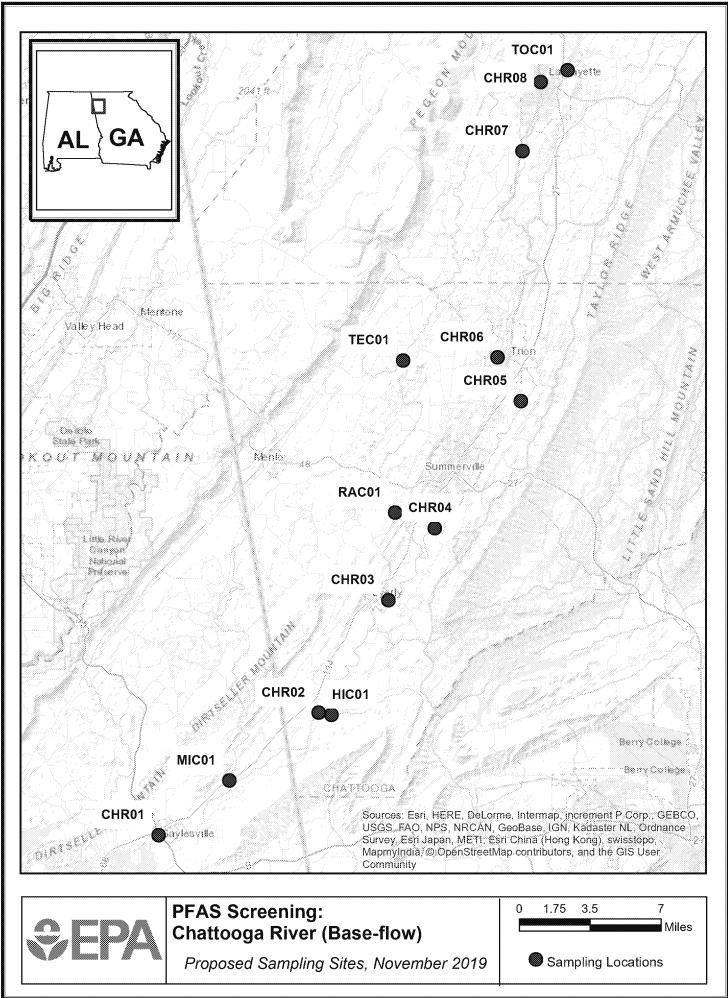
Method Detection Limits (MDLs) & Minimum Reporting Limits (MRLs)					
Analyte <sup>1</sup>		iter² (ppb)	Soil/Sediment³ μg/kg (ppb)		
	MDL	MRL	MDL	MRL	
Perfluorotridecanoic acid (PFTrDA)	0.039	0.040	0.040	0.100	
Perfluorododecanoic acid (PFDoA)	0.029	0.040	0.040	0.100	
Perfluoroundecanoic acid (PFUDA)	0.021	0.040	0.040	0.100	
Perfluorodecanoic acid (PFDA)	0.096	0.160	0.040	0.100	
Perfluorononanoic acid (PFNA)	0.016	0.040	0.040	0.100	
Perfluorooctanoic acid (PFOA)	0.026	0.040	0.040	0.100	
Perfluoroheptanoic acid (PFHpA)	0.014	0.040	0.040	0.100	
Perfluorohexanoic acid (PFHxA)	0.031	0.040	0.040	0.100	
Perfluoropentanoic acid (PFPeA)	0.018	0.040	0.040	0.100	
Perfluorobutyric acid (PFBA)	0.022	0.040	0.040	0.100	
Perfluorodecanesulfonate (PFDS)	0.032	0.039	0.040	0.096	
Perfluorononanesulfonate (PFNS)	0.015	0.038	0.040	0.096	
Perfluorooctanesulfonate (PFOS)	0.017	0.037	0.040	0.092	
Perfluoroheptanesulfonate (PFHpS)	0.017	0.038	0.040	0.095	
Perfluorohexanesulfonate (PFHxS)	0.017	0.036	0.040	0.091	
Perfluoropentanesulfonate (PFPeS)	0.013	0.038	0.040	0.094	
Perfluorobutanesulfonate (PFBS)	0.023	0.035	0.040	0.088	
Perfluorooctanesulfonamide (FOSA)	0.031	0.040	0.040	0.100	
Fluorotelomer sulfonate 8:02 (8:2 FTS)	0.034	0.038	0.040	0.096	
Fluorotelomer sulfonate 6:02 (6:2 FTS)	0.029	0.038	0.040	0.095	
Fluorotelomer sulfonate 4:02 (4:2 FTS)	0.021	0.037	0.040	0.094	
N-(Heptadecafluorooctylsulfonyl)-N-methylglycine (N-MeFOSAA)	0.110	0.160	0.040	0.100	
Hexafluoropropylene oxide-dimer acid (HFPO-DA)	0.026	0.040	0.040	0.100	

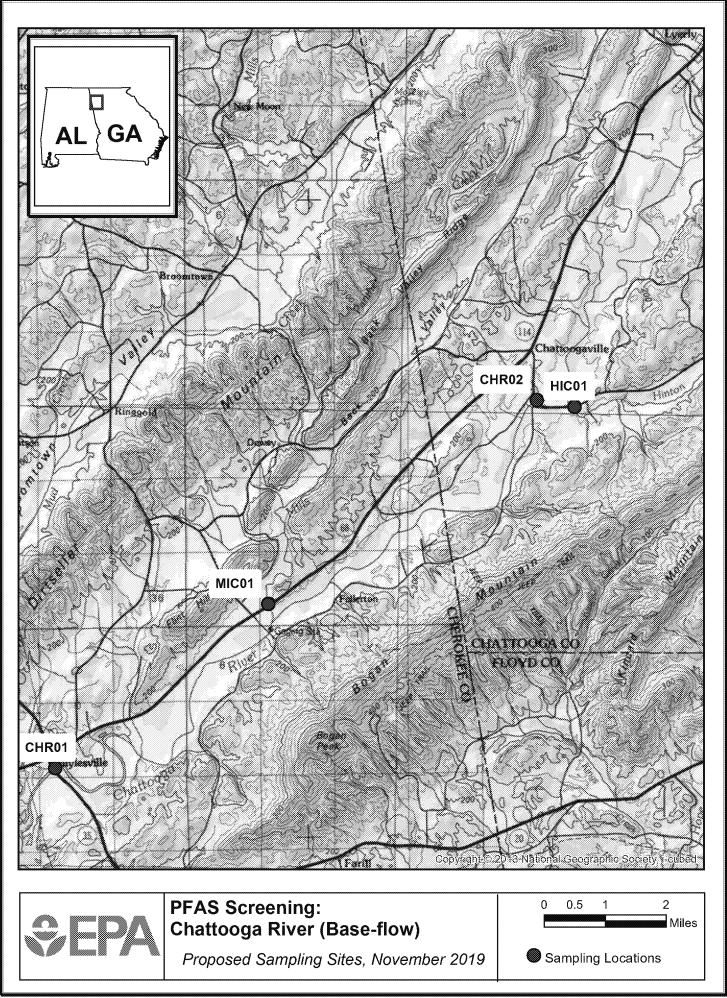
<sup>&</sup>lt;sup>1</sup>PFAS analytes for both surface water and sediment/soil matrices are analyzed via the method outlined in LSBPROC-800-R1.

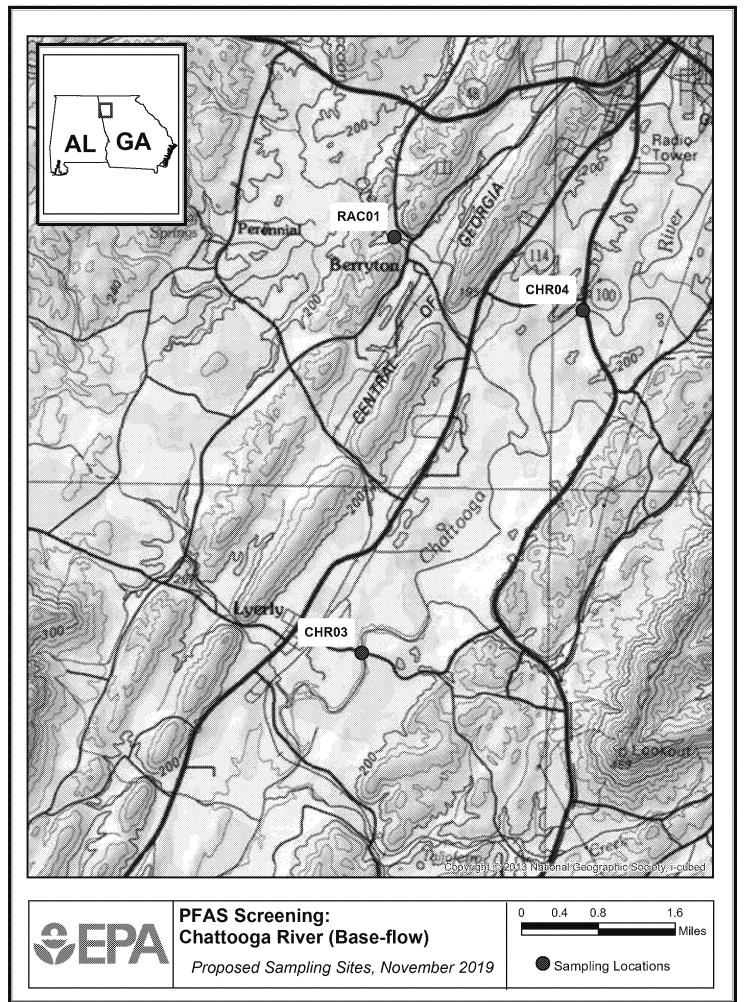
<sup>&</sup>lt;sup>2</sup>PFAS analytes in surface water are analyzed using ASTM standard D7979-17.

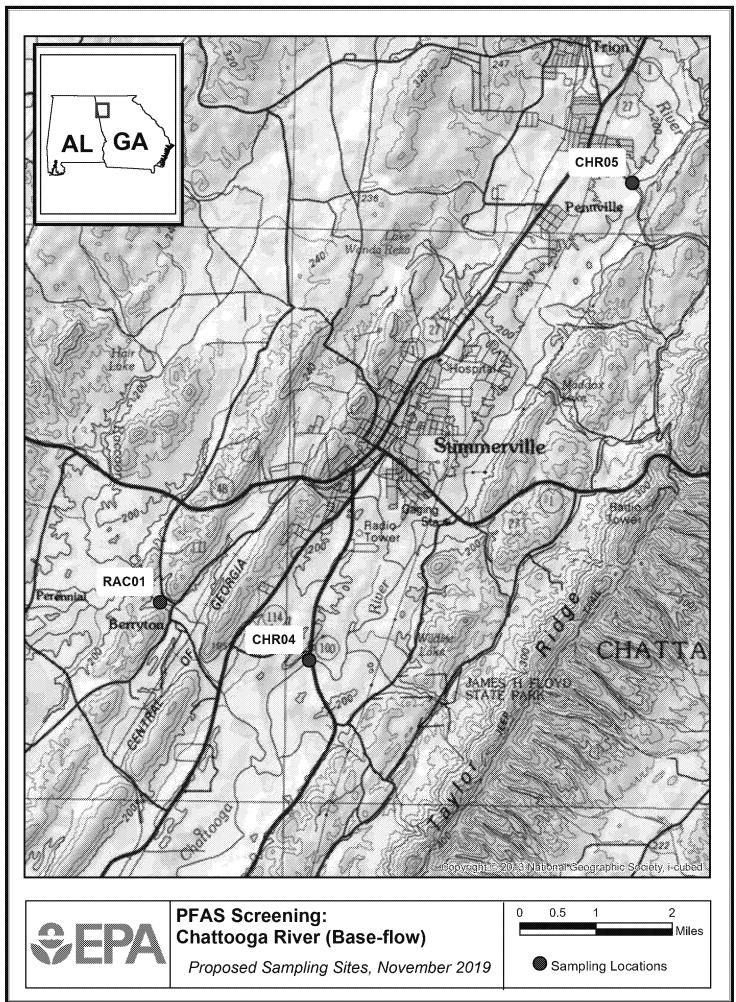
<sup>&</sup>lt;sup>3</sup>PFAS analytes in solids (e.g. soil, sediment, and waste) are analyzed using ASTM standard D7968-17a.

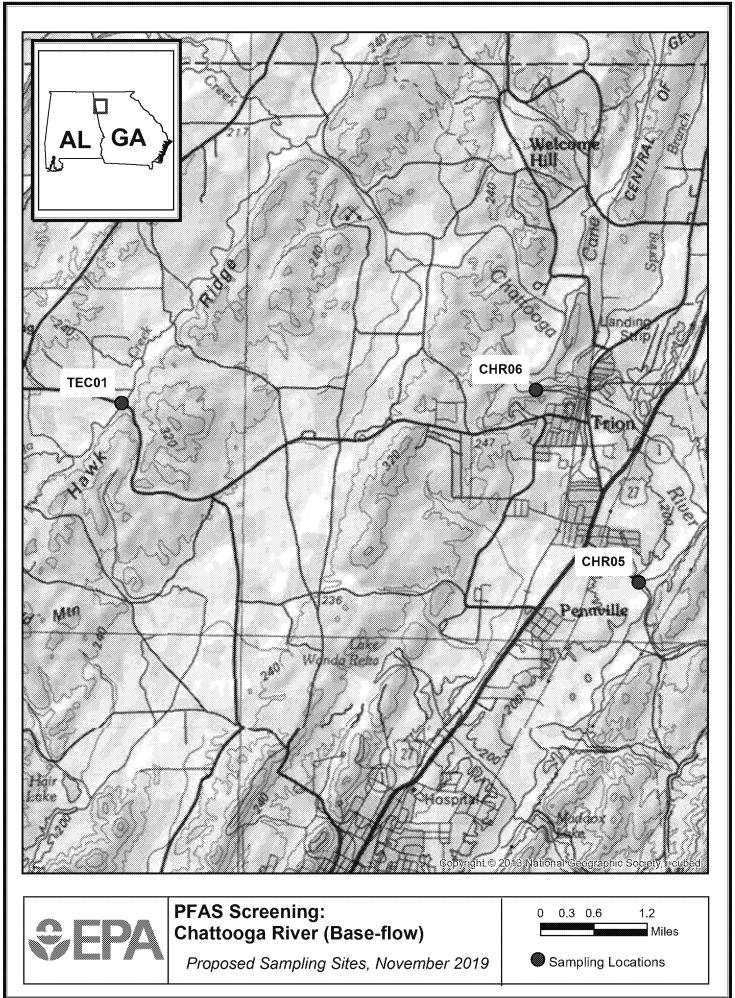
**Appendix A: Site Maps** 

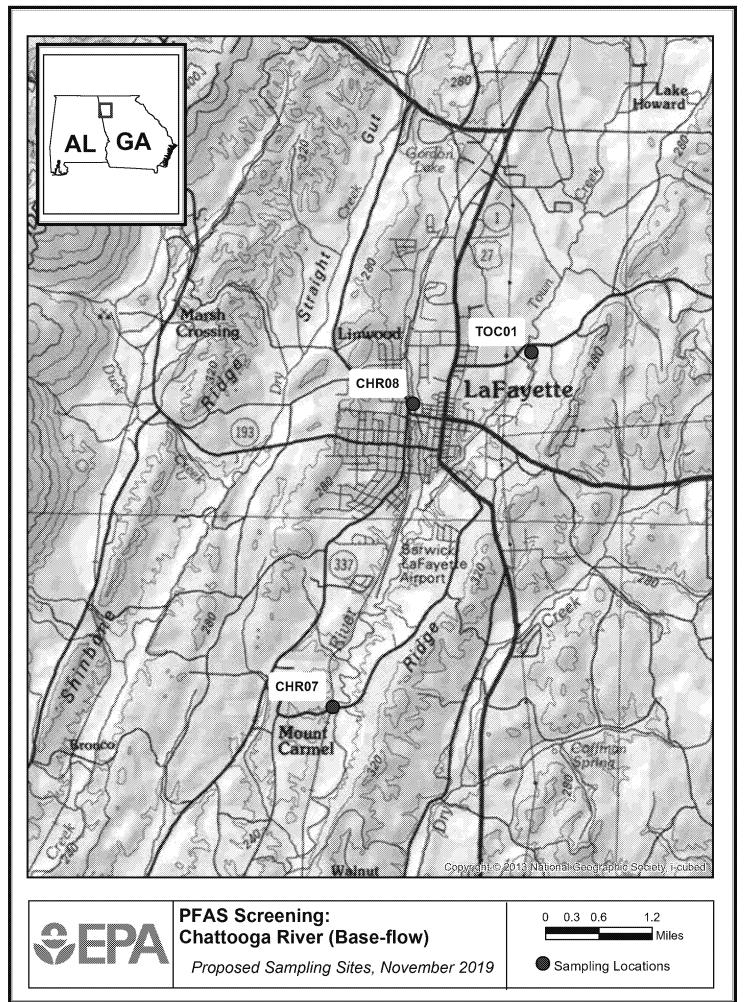












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